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RECOMMENDED SPECIFICATION FOR QUICKLIME AND HYDRATED LIME FOR USE IN THE MANUFACTURE OF SAND-LIME BRICK.

This is the sixth of a series of specifications for the lime used in various chemical industries. To assist in the development of these specifications the bureau has called together an Interdepartmental Conference on Chemical Lime, composed of representatives of the Geological Survey and Bureau of Mines of the Interior Department; the Bureau of Soils, Bureau of Chemistry, Forest Service, and Fixed Nitrogen Research Laboratory of the Department of Agriculture; and the Chemical Warfare Service of the War Department. The present specification, based on a draft originally prepared by W. E. Emley of the lime section, Bureau of Standards, has been unanimously approved by the above conference and by the National Lime Association.

ABSTRACT.

Sand-lime brick is made of sand bound together with lime. The lime is caused to combine chemically with some of the sand by treatment with high-pressure steam. Lime for this purpose must be completely hydrated and must be reasonably free from magnesia and carbon dioxide. About 85 per cent purity is required.

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1. GENERAL.

(a) **USE OF LIME IN MAKING SAND-LIME BRICK.**—Sand-lime brick is made by mixing sand and lime, pressing the mixture into brick form, and curing with high-pressure steam. The steam causes a chemical reaction to occur between the lime and sand, forming what is believed to be a hydrated calcium silicate. This silicate acts as a binding material to hold together the excess sand, which forms the major portion of the brick.

The lime must be completely hydrated before it is mixed with the sand and made into brick. Otherwise, subsequent hydration is apt to weaken or disrupt the brick. Overburned lime or magnesia may cause trouble because they hydrate slowly.

Most manufacturers of sand-lime brick prefer, for economical reasons, to buy quicklime and make their own hydrate. Others buy commercial hydrated lime.

(b) **DEFINITIONS.**—Quicklime is the product resulting from the calcination of limestone and consists essentially of calcium oxide, or of calcium and magnesium oxides. It will slake when water is added to it, and this slaking is accompanied by an evolution of heat and an increase in volume. Hydrated lime is a dry powder which is made by treating quicklime with enough water to satisfy its chemical affinity under the conditions of manufacture. It consists essentially of calcium hydroxide, or of a mixture of calcium hydroxide and magnesium oxide or hydroxide.

(c) **PACKING.**—Quick lime is shipped either in bulk in carload lots or barrels holding 180 pounds net or 280 pounds net each. Hydrated lime is shipped in paper bags holding 50 pounds net each.

2. REQUIREMENTS.

(a) **QUALITY.**—Quicklime must be of such a nature that it will slake freely and completely. It must, therefore, be free from ashes, clinker, fused silicates, and similar visible impurities. Hydrated lime must be completely hydrated. Such chemical impurities as silica, iron, and alumina may be regarded merely as diluents, except when they are fused with the lime to form slowly slaking compounds.

(b) **COMPOSITION.**—Quicklime or hydrated lime shall contain not less than 85 per cent calcium oxide nor more than 5 per cent magnesium oxide, both figures being based on the nonvolatile portion of the material.

The carbon dioxide in the quicklime as received shall not be more than 5 per cent. The carbon dioxide in the hydrated lime as received shall not be more than 7 per cent.

Hydrated lime shall contain sufficient water to meet the chemical requirements of the calcium oxide.

(c) MARKING.—Each carload of material, or fraction thereof, shall be legibly marked with the names of the consignor and consignee, and with some means of identifying the particular contract on which the shipment is made.

This information is in addition to that required by the Federal lime-barrel law.

(d) RETESTING.—Notice of the rejection of a shipment based on these specifications must be in the hands of the consignor within 10 days after the receipt of the shipment at the point of destination. If the consignor desires a retest, he shall notify the consignee within five days of receipt of the notice of rejection. The consignee shall provide all reasonable facilities to permit the consignor to resample the material. This retest shall be at the expense of the consignor.

3. SAMPLING AND TESTING.

(a) SAMPLING.—The purchaser shall bear all expense of sampling and testing. When quicklime is shipped in bulk, the sample shall be so taken that it will represent an average of all parts of the shipment from top to bottom, and shall not contain a disproportionate share of the top and bottom layers, which are most subject to changes. The sample shall consist of 1 shovelful for each 3 tons of material, but not less than 10 shovelfuls, taken from different parts of the shipment. The total sample taken shall weigh at least 100 pounds, shall be mixed thoroughly, and "quartered" to provide a 15-pound sample for the laboratory. In case a shipment consists of more than one car, a separate sample shall be taken from each car.

When quicklime is shipped in barrels, at least 3 per cent of the number of barrels shall be sampled. They shall be taken from various parts of the shipment, dumped, mixed, and sampled as specified in the above paragraph.

In the case of hydrated lime, the sample shall be a fair average of the shipment. Three per cent of the packages shall be sampled. The sample shall be taken from the surface to the center of the package. The material so obtained shall be thoroughly mixed and quartered to provide a 2-pound sample for the laboratory.

When sampling quicklime or hydrated lime, it is essential that the operation be conducted as expeditiously as possible, in order to avoid undue exposure of the material to the air. The sample to be sent to the laboratory shall immediately be placed in an air-tight container in which the unused portion shall be stored until the shipment has been finally accepted or rejected by the purchaser.

The sample may be taken either at the point of shipment or at the point of destination, as agreed upon by the contracting parties.

(b) TESTING.—The following directions for analytical work are taken from American Society for Testing Materials, Tentative Methods of Chemical Analysis of Limestone, Quicklime, and Hydrated Lime, C25-22T. For further information on this subject, reference should be made to "The analysis of silicate and carbonate rocks," by W. F. Hillebrand, U. S. Geological Survey, Bulletin No. 700.

Weigh out 0.5 g of the substance and, if hydrated lime, ignite in a covered platinum crucible in an electric muffle for 15 minutes, or longer if the heat is not powerful enough to effect complete decomposition within that time. Transfer to an evaporating dish, preferably of platinum for the sake of celerity in evaporation, mix to a thin slurry with distilled water, add 5 to 10 cc of HCl (sp. gr. 1.20) and digest with aid of gentle heat and agitation until solution is complete. Solution may be aided and the time shortened by light pressure with the flattened end of a glass rod upon resistant lumps. Then evaporate the solution to dryness, so far as this may be possible on the water bath.

When dry or nearly so, place the dish and its contents in an air bath or (covered) on a platinum triangle resting on a hot plate. Drench the cooled mass with HCl (sp. gr. 1.20) and allow to stand for a few minutes. Add an equal volume of water, cover the dish, and place on the water bath for 10 minutes. Separate the silica by filtration on a filter of suitable size. Wash thoroughly with dilute HCl and then wash twice with cold water.

Evaporate the filtrate to dryness. Extract the residue with HCl as before, but allowing only a few minutes time. Filter this solution through a second and smaller paper.

Transfer, wet, the papers containing the separated residue to a platinum crucible. Char carefully without allowing the paper to inflame and ignite in an electric muffle and weigh. Correct the silica for foreign matter by evaporating with 5 cc of HF and one or two drops of H_2SO_4 . Heat the residue for two or three minutes in an electric muffle.

Fuse the residue from the silica determination using a little Na_2CO_3 . Dissolve the cooled melt in HCl and add the solution to the filtrate from the second evaporation for silica. Add a few drops of bromine water or HNO_3 and boil the solution until all trace of bromine or chlorine is gone. Then add HCl, if not already present, sufficient to insure a total volume of 10 to 15 cc of HCl (sp. gr. 1.20). Add a few drops of methyl red solution, and after diluting to 200 to 250 cc, heat the liquid to boiling. Neutralize by means of NH_4OH (dilute toward the end) until the color of the liquid changes to a distinct yellow. Boil for one or two minutes, allow to settle, filter and wash the precipitate at once two or three times with hot NH_4Cl (2 per cent) solution, and suck dry.

Set the filtrate aside. Dissolve any precipitate from the paper in hot, dilute HCl, the solution passing into the beaker in which the precipitation was made and wash the

paper thoroughly with hot water. Boil the solution to expel any trace of chlorine and again precipitate the hydroxides with NH_4OH as described above.

Add a few drops of NH_4OH to the combined filtrate from the R_2O_3 precipitate, and bring the solution to boiling. To the boiling solution add 35 cc of a saturated solution of $(\text{NH}_4)_2\text{C}_2\text{O}_4$, and continue the boiling until the precipitated CaC_2O_4 assumes a granular form; then allow it to stand 20 minutes, or until the precipitate has settled and the supernatant liquid is clear, filter and wash thoroughly with boiling water. Place the wet filter and precipitate in a platinum crucible, and burn off the paper over the small flame of a Bunsen burner, or in a muffle. Ignite the residue, dissolve in hot dilute HCl , and make up the volume of the solution to 100 cc with water. Add NH_4OH in slight excess and boil the liquid. If a small amount of $\text{Al}_2(\text{OH})_6$ separates, filter it out, wash with NH_4Cl (2 per cent solution), ignite, weigh, and add this weight to that found in the first determination. Then reprecipitate the lime by $(\text{NH}_4)_2\text{C}_2\text{O}_4$, allow to stand until settled, filter, wash thoroughly with boiling water, and weigh as calcium oxide after ignition to constant weight in a covered platinum crucible.

Acidify the combined filtrates from the calcium precipitates with HCl and concentrate on the water bath to about 150 cc, and heat to boiling. To the boiling solution add 10 cc of a saturated solution of $\text{Na}(\text{NH}_4)\text{HPO}_4$ and continue the boiling for several minutes. Then cool to room temperature, and add NH_4OH drop by drop with constant stirring until the crystalline ammonium-magnesium orthophosphate begins to form and then in moderate excess, the stirring being continued for several minutes. Allow the liquid to stand in a cool atmosphere for from 12 to 48 hours and filter.

Dissolve the precipitate in hot dilute HCl , make up the solution with water to about 100 cc, add 1 cc of the saturated solution of $\text{Na}(\text{NH}_4)\text{HPO}_4$ and NH_4OH drop by drop with constant stirring until the precipitate is again formed as above described and the ammonia is present in moderate excess. Then allow it to stand in a cool atmosphere for from 12 to 48 hours, filter on paper or a Gooch crucible, wash with dilute NH_4OH containing NH_4NO_3 , ignite, cool, and weigh as $\text{Mg}_2\text{P}_2\text{O}_7$.

Place 1 g of the properly prepared sample in a weighed platinum crucible, cover with the lid and heat gently for 5 minutes, gradually increasing the temperature to the maximum of the muffle, and maintain at this temperature until constant weight is obtained (usually about 15 minutes).

Carbon dioxide is to be determined upon the properly prepared sample according to the method given in U. S. Geological Survey Bulletin No. 700, page 217. An illustration showing the arrangement of the component parts of the necessary apparatus is shown on page 218 of that bulletin.

Boil the 5 g sample with dilute HCl in a small Erlenmeyer flask attached to an upwardly inclined condenser, whence, after passing through a drying system—calcium chloride, anhydrous copper sulphate to retain hydrogen sulphide from decomposable sulphides and any HCl that may pass over, then calcium chloride again—carbon dioxide is caught by absorption tubes filled with soda lime followed by calcium chloride. Of course, arrangement is made for a current of air free from CO_2 with which to sweep out the apparatus before and after the experiment and for a slow current during its continuance.

The manipulations are as follows: Pour hot water upon the powder in the flask fitted with a separatory funnel and delivery tube; attach this to the condenser, and force a current of air free from CO_2 through the whole system, except the weighed absorption tubes, until the original air has been displaced, the observation bulbs (containing H_2SO_4 to show the rate of gas flow) being attached directly to the drying system. Then close the stopcock in the separatory funnel, half fill the latter with HCl (1 : 1), replace the rubber stopper of the funnel, insert the absorption tubes between the drying system and the observation bulbs, and allow the acid to flow into

the flask, slowly if there is much carbon dioxide, rapidly if there is but little. When effervescence diminishes in the former case, at once in the latter, light the burner under the flask and start the flow of water through the condenser. Keep the flame low, so as to secure steady but quiet ebullition, and do not interrupt the air current although it should be reduced to a slow rate. With much carbon dioxide the rate of absorption is very readily noted by holding the hand to the soda-lime tubes, which become hot or warm when the absorption is taking place. Sufficient time having elapsed, extinguish the flame, and increase the air current. When cool, disconnect the soda-lime tubes from the apparatus and allow to stand in the balance case until two weights taken 30 minutes apart agree within 0.5 mg.

The soda lime for use in this determination must be porous, not hard and unabsorptive like that sometimes used for the combustion of nitrogenous organic substances.

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